



Physico-chemical, mechanical, microstructure and durability characteristics of alkali activated Egyptian slag



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HIGHLIGHTS

- Alkali activator contents affected the location and intensities of the conductivity maximum.
- Mix S5 shows the higher values of compressive strength at all curing ages up to 90 days.
- Mix S1 shows higher values of strength in 5% MgSO₄ or MgCl₂ solution upto 180 days.
- Alkali activated GBFS improves the durability and the resistance to chemical attack.
- The total chloride and total sulphate contents decrease with alkali activated GBFS.

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ABSTRACT

The aim of the present work is to study the characteristics of alkali activated Egyptian slag (GBFS) mixed with different contents of Na₂O and SiO₂. The rate of the kinetic of activation of GBFS has been studied by electrical conductivity, FTIR, DTA/TGA, XRD and SEM techniques. Electrical conductivity of alkali activated slag systems depends mainly on the binder composition, the activator type and its concentration. The increase in alkali activator contents affected the location and intensities of the conductivity maximum. As the hydration time increases the amount of hydration products increase, hence the chemically combined water and combined slag contents increase. SEM images showed the presence of (N,C)ASH gel with low porosity. With the increase of the content of Na₂O leads to form a denser closed microstructure, leading to higher compressive strength values. The activated GBFS showed good durability in 5% MgSO₄ or 5% MgCl₂ solution, i.e., the compressive strength increased gradually with immersing time up to 180 days. The total chloride and total sulphate contents decrease with alkali activated GBFS due to the forming hydrated products that fill some available open pores, thereby inhibiting Cl⁻ or SO₄²⁻ ions penetration; this effect leads to a decreased accessibility of Cl⁻ or SO₄²⁻ ions towards the more dense with low capillary pore structure. It can be concluded that alkali activated GBFS are more durable in 5% MgSO₄ or 5% MgCl₂ than OPC pastes.

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1. Introduction

The role of alkali activated cements protect the environment through the utilization of by-product materials in their manufacture and end applications in waste management. The production of alkali-activated concretes is associated with low energy consumption and low CO₂ emission, along with the potential to reach high mechanical strength at early ages of curing, resistance

to chemical attack and resistance to high thermally treated temperatures. These properties have made concretes based on alkali-activated binders a very interesting [1–3]. Portland cement (OPC) production increases global greenhouse gas emissions such as dioxin, NO_x, SO₂, and particulates, through the calcination of clinker in hydrocarbon heated furnaces. The high CO₂ emissions arising (7% of all CO₂ generated) from OPC manufacture have been attributed to calcination of limestone, which leads to formation and release of CO₂ and high energy consumption during manufacturing at temperatures up to 1550 °C [4]. Reduction in cement consumption has been attained by the use of industrial by products such as blast-furnace slag (GBFS), fly ash (FA), silica fume (SF) and cement kiln dust (CKD), as partial cement replacement materials [5,6].

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Alkali activation is a process based on the action of solutions of alkali compounds like sodium or calcium hydroxide, sodium carbonate and silicate on the aluminosilicate constituent of the binder. The result is the destruction of the chemical bonds of the type Si—O—Si in the aluminosilicate. Alkali-activated binders (also referred as to ‘geopolymers’) are produced from an aluminosilicate source mixed with an alkaline solution [7]. These binders exhibit technological and ecological advantages in comparison with ordinary Portland cement (OPC), and the industrial use of geopolymer concretes in the production of high performance building materials is expanding [8]. A strong dependence on the nature of the alkaline activator in the structural and mechanical development of these binders has been identified throughout the literature. Alkali activated cements (AAC) are the type of the binders synthesized from natural aluminosilicate materials and aluminosilicate waste products such as fly ash, rice husk ash, and slag which are rich in silicon and aluminium [9]. Therefore, the use of alkali activation technology enables the possibility of substantial utilization of waste materials.

Development of geopolymers is the new, knowledge-based, multi-functional materials with higher performance, reduced environmental impact and adjustable to customer needs. Numerous studies have been conducted over the last few decades to determine the composition and microstructure property relationships in such systems [10–14]. Effect of workability, ultra fine materials, pore size distribution, early age strength, mechanical, cracking and shrinkage as well as the residual compressive strength behaviour of alkali activated slag paste (AASP) up to 1200 °C were investigated [15–22]. The workability, durability and strength are affected by the type and concentration of the alkaline activator, solution ratio ($M_s = \text{SiO}_2/\text{Na}_2\text{O}$), slag type, fineness, curing conditions, water/cement ratio, activating solution/slag ratio and the use of admixture as well as fibres [23–26]. Purdon [27] investigated the effect of NaOH concentration on strength of AAC. Glukhovskiy et al. [28] classified alkaline activators into six groups according to their chemical compositions as a caustic alkalis, non-silicate weak acid salts, silicates, aluminates, aluminosilicates and non-silicate strong acid salts. NaOH, Na_2CO_3 , $\text{Na}_2\text{O} \cdot n\text{SiO}_2$ and Na_2SO_4 are the most widely available and economical activators. The factors affecting on the strength of alkali activated GBFS were the type, and the dosage of alkali as well as the type and fineness of slag, $\text{SiO}_2/\text{Na}_2\text{O}$ ratio, curing temperature, water/slag ratio and additive as well as [29,30].

The effectiveness of the alkali activator is a very significant factor influencing the intensity and rate of process of alkali activation. It is reported that the best activator is waterglass [31,32]. The best dosage is within the range of 3.0–5.5% Na_2O by of slag. The optimum dosage ranges of waterglass solution are 0.75–1.25% for acid GBFS, 0.90–1.30% for neutral GBFS and 1.0–1.5% for basic GBFS. The optimum range of fineness is 4000–5500 cm^2/g . It is evident that the effectiveness of activator is a very significant factor which determines the engineering properties of the material. Sodium silicate (SSL) has been extensively used in the activation of aluminosilicate precursors such as GBFS, metakaolin (MK), FA and others [23,33].

The sulphate and chloride ions can enter into deleterious reactions leading to the dissolution of CH, precipitation of gypsum and sulfoaluminates as well as Friedel’s salt, which cause expansion and softening of concrete, respectively [34,35]. The presence of Mg^{2+} as MgSO_4 and/or MgCl_2 leads to the distortion of C—S—H through a dissolution of Ca^{2+} and/or an exchange with Mg^{2+} to give also Mg—S—H and CH. All the above mentioned reactions are accompanied by decrease in strength [36–40]. El-Didamony et al. [41] represented the experimental trials to activate GBFS to produce cementless binding materials. The results revealed that the increase of NaOH content increase the chemically combined water

contents up to 90 days. On the other hand, the bulk density and compressive strength were increased by increasing Na_2SiO_3 content in presence of NaOH. The increase of compressive strength is due the increase of $[\text{SiO}_4]^{4-}$ which enhances the C—S—H formation. The durability of alkali activated GBFS mortars and concretes have been studied. Alkali activated GBFS are durable binder in sulphate, sea water and acid resistant [42–48]. Darko and Branislav [49] examined the early hydration of alkali-GBFS metasilicate ($\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$) solution at 25 °C. The compressive strength of AAC is higher than OPC mortar. The feasibility of using an alkaline activated GBFS to produce a mortar without using OPC was investigated [50].

The reaction mechanism of aluminosilicates containing a calcium bearing compound differs from the geopolymeric reaction as explained. It has also been reported that the type of calcium bearing compound in the starting material play an important role in the alkali activation of these materials. Alkalis first attack the GBFS particles breaking the outer layer and then a polycondensation of reaction products takes place. Wang et al. [51] suggested that the initial reaction products formed by the dissolution and precipitation, whereas, at later ages, a solid state mechanism is followed where the reaction takes place on the surface of the formed particles, dominated by slow diffusion of the ionic species into the unreacted core. Alkali cation (R^+) acts as a mere catalyst for the reaction in the initial stages of hydration, via cation exchange with the Ca^{2+} ions [52,53]. The alkaline cations act as structure creators. The nature of the anion in the solution also plays a determining role in activation, particularly in early ages and especially with regard to paste setting [47,54]. The final products of the GBFS reaction are similar to the products of cement hydration (C—S—H); the major difference being the rate and intensity of the reaction. It has also been observed that the alkalis are bound to the reaction products and are not freely available in the pore solution (this depends on the alkali concentration used, though), thereby a small percentage of the alkalis (around 0.02%) are bound to the reaction products but most of the alkalis remains in the pore solution [55].

This paper studied the influence of different alkaline concentrations on the hydration characteristics and morphology of GBFS system. These characteristics were investigated by changing the initial alkalinity of the hydration condition in order to reveal the affecting mechanism of alkaline condition on its hydration.

2. Experimental techniques

2.1. Materials

Egyptian blast-furnace slag (GBFS) was provided from Iron and Steel Company, Helwan Governorate, Egypt. Its chemical oxide composition is given in Table 1. XRD patterns, FTIR spectra and SEM are shown in Figs. 1–3. Blaine surface area of slag was $4500 \pm 50 \text{ cm}^2/\text{g}$. The diffraction patterns of GBFS was shown in Fig. 1. The diffractogram of the unreacted slag shows a predominantly amorphous material. There is also a broad feature ‘amorphous hump’ between 20 and 38° 2θ attributed to the amorphous glassy component.

Table 1
Chemical composition of starting materials, mass %.

Oxide composition (%)	GBFS	OPC	SSL
SiO_2	37.81	21.40	30.7
Al_2O_3	13.14	3.67	–
Fe_2O_3	0.23	5.05	–
CaO	38.70	64.03	–
MgO	7.11	1.50	–
SO_3	1.19	2.05	–
Na_2O	1.03	0.30	10.3
K_2O	0.19	0.22	–
TiO_2	0.34	–	–
P_2O_5	0.16	–	–
L.O.I.	0.00	1.60	59.0
Total	99.90	99.82	100.0

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